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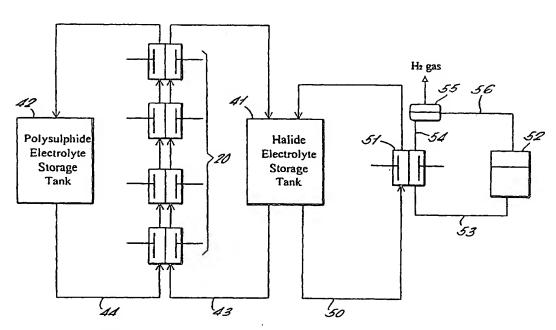
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(54) Title: ELECTROLYTE REBALANCING SYSTEM



(57) Abstract: A process for rebalancing the electrolyte system in a regenerative fuel cell using a sulfide/polysulfide reaction in one half of the cell and a bromine/bromide reaction in the other half of the cell comprises passing the electrolyte containing sulfide/polysulfide or bromine/bromide through the +ve chamber of an auxiliary cell and passing an electrolyte containing water and being free from polysulfide or bromine through the -ve chamber of the auxiliary cell, the auxiliary cell operating so as to oxidise sulfide ions to sulfur or bromide ions to bromine in the +ve chamber and to reduce water to hydrogen and hydroxide ions in the -ve chamber.

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For two-letter codes and other abbreviations, refer to the "Guidance Notes on Codes and Abbreviations" appearing at the beginning of each regular issue of the PCT Gazette.

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Electrolyte Rebalancing System

The present invention relates to the field of regenerative fuel cell (RFC) technology. In

5 particular it relates to apparatus and methods for the operation of RFCs which enhance their performance characteristics.

The manner in which RFCs are able to store and deliver electricity is well known to those skilled in the art. An example of an RFC is described in US-A-4485154 which discloses an electrically chargeable, anionically active, reduction-oxidation system using a sulfide/polysulfide reaction in one half of the cell and an iodine/iodide, chlorine/chloride or bromine/bromide reaction in the other half of the cell. The two halves of the cell are separated by a cation exchange membrane.

The overall chemical reaction involved, for example, for the bromine/bromide-sulfide/polysulfide system is shown in Equation 1 below:

$$Br_2 + S^{2-} \rightleftharpoons 2Br^{-} + S$$
 Equation 1

However, within an RFC such as that described in US-A-4485154, the reaction takes place in separate but dependent bromine and sulfur half-cell reactions as shown below in Equations 2 and 3:

$$Br_2 + 2e^- = 2Br^-$$
 Equation 2
 $S^{2-} = 2e^- + S$ Equation 3

It should be noted however that these equations represent the overall reactive changes occurring in the RFC. In practice the reactions are complicated by

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the low basicity of sulfide which results in the formation of bisulfide as the active species, as shown in Equation 4.

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 $S^{2-} + H_2O = HS^- + OH^-$ Equation 4

Also, the sulfur produced in Equations 1 and 3 forms soluble polysulfide species in the presence of sulfide ions, as shown in Equation 5 (where x may be from 1 to 4).

 $S^{2-} + xS = S_{x+1}^{2-}$ Equation 5

Also, free bromine is solubilised in the presence of bromide ions to form the tribromide ion, as shown in Equation 6

 $Br^{-} + Br_{2} \Rightarrow Br_{3}^{-}$ Equation 6

When the RFC is discharging, bromine is converted to bromide on the +ve side of the membrane and sulfide is converted to polysulfide on the -ve side of the membrane. Equation 1 goes from left to right and metal ions flow from the -ve side of the membrane to the +ve side of the membrane to complete the circuit. When the RFC is charging, bromide is converted to bromine on the +ve side of the membrane and polysulfide is converted to sulfide on the -ve side of the membrane. Equation 1 goes from right to left and metal ions flow from the +ve side of the membrane to the -ve side of the membrane to complete the circuit.

The discharge/charge cycle described above will be repeated many times during the lifetime of the RFC and in order for the RFC to work efficiently throughout its lifetime it is important that the electrolytes remain balanced. In the context of the present

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specification, when the term "balanced" is used to describe the electrolytes it means that the relative concentrations of the reactive species within the electrolytes are maintained at, or close to, values which enable optimum performance of the RFC.

Similarly, in the context of the present specification, the term "rebalancing" refers to a process which alters the concentration of one or more reactive species in one or both of the electrolytes so as to return said electrolytes to a balanced state or so as to maintain said electrolytes in a balanced state.

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At the beginning of the RFC's lifetime the relative concentrations of the reactive species on either side of the membrane will normally be fixed so that the electrolytes are balanced. However, once the RFC begins to operate in its repeating discharge-charge cycle, factors may intervene which result in the electrolytes becoming unbalanced. These factors will vary depending upon the identity of the reactive species within the electrolytes and on the manner in which the RFC is constructed and operated.

25 In the case of the bromine/bromide-sulfide/polysulfide RFC such as that described above, the most important factor which results in the electrolytes becoming unbalanced is the diffusion of unwanted species across the membrane. Although a cation selective ion-30 exchange membrane is used, 100% permselectivity is not possible and during extended cycling of the cell some anionic species diffuse through the membrane. particular, sulfide ions (largely present in the bisulfide form, HS⁻) and polysulfide ions $(S_{**}, {}^{2}, where$ 35 x may be from 1 to 4) may diffuse from the sulfide/polysulfide electrolyte into the bromine/bromide electrolyte where they will be

oxidised by the bromine to form sulfate ions as shown in equations 7 and 8 below:

$$HS^{-} + 4Br_{2} + 4H_{2}O - 8Br^{-} + SO_{4}^{2-} + 9H^{+}$$

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Equation 7

$$S_{x+1}^{2-}$$
 + (3x+4)Br₂ + (4x+4)H₂O -

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 $(6x+8)Br^{-} + (x+1)SO_{4}^{2-} + (8x+8)H^{+}$

Equation 8

Imperfections other than diffusion through the

membrane which could similarly contribute to the above process are ineffective sealing between cell compartments, or catastrophic failure of any of the cell separating components, each of which may result in crossover of the electrolytes between cell compartments.

In Equations 7 and 8, the oxidation of the sulfur species goes beyond that which occurs during normal operation of the RFC. That is to say, the sulfide and polysulfide ions are oxidised all the way to sulfate ions. Consequently, in the case of sulfide ion crossover (Equation 7), four bromine molecules per sulfide ion are consumed rather than the normal one bromine molecule per sulfide ion which is consumed in the reaction scheme of Equation 1. Similar overconsumption of bromine results from polysulfide cross-over (Equation 8) although to a slightly lesser extent. As a result, the bromine/bromide electrolyte becomes discharged to a greater extent than the sulfide/polysulfide electrolyte. Thus, when the cell is discharging there is insufficient bromine present to react with all the sulfide ions present thereby

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preventing completion of the discharge cycle. As a result, the voltage generated by the cell begins to decline earlier in the discharge cycle than when the electrolytes are balanced. In effect, the reactions represented by Equations 7 and 8 result in the conversion of some of the polysulfide ions to sulfide because not all of the polysulfide ions are recovered on discharge. Subsequent cycles repeat this process, further reducing the number of polysulfide ions present. Ultimately, there will be insufficient polysulfide ions present to accept charge during the charge cycle. Since the electrochemistry has to continue if charging is maintained, the next most favourable reaction occurs, i.e. water is reduced and the electrode on the -ve side of the cell starts to gas hydrogen.

It would therefore be advantageous to provide a process for rebalancing the electrolytes in order to compensate for the unbalancing effect of the crossover of sulfide and/or polysulfide electrolyte species into the bromine electrolyte. Although it would be possible to replace the electrolytes in the system with fresh electrolytes at periodic intervals, this is disadvantageous because of the economic implications and because of the environmental implications of the great amounts of waste electrolytes which would require to be disposed of.

- Accordingly, the present invention provides an electrochemical process for energy storage and/or power delivery comprising:
 - (i) maintaining and circulating electrolyte flows in a fully liquid system in which the active constituents are fully soluble in a single cell or in an array of repeating cell structures, each cell with a positive (+ve) chamber containing an

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inert +ve electrode and a negative (-ve) chamber containing an inert -ve electrode, the chambers being separated from one another by a cation exchange membrane, the electrolyte circulating in the -ve chamber of each cell during power delivery containing a sulfide (electrolyte 1), and the electrolyte circulating in the +ve chamber during power delivery containing bromine (electrolyte 2),

- (ii) restoring or replenishing the electrolytes in the +ve and -ve chambers by circulating the electrolyte from each chamber to storage means comprising a volume of electrolyte greater than the cell volume for extended delivery of power over a longer discharge cycle than the cell volume alone would permit, and
- (iii) rebalancing the electrolytes by circulating a fraction of electrolyte 1 or electrolyte 2 through the +ve chamber of an auxiliary cell, 20 said auxiliary cell comprising a +ve chamber containing an inert +ve electrode and a -ve chamber containing an inert -ve electrode, the chambers being separated from one another by a cation exchange membrane, the electrolyte circulating through the -ve chamber of the 25 auxiliary cell containing water and being free from polysulfide and free from bromine during rebalancing, the auxiliary cell operating so as to oxidise sulfide ions to sulfur or bromide ions 30 to bromine in the +ve chamber and so as to reduce water to hydrogen and hydroxide ions in the $-^{ve}$ chamber.

The oxidation of bromide to bromine rebalances the electrolytes by restoring the bromine which is reduced by reaction with migrating sulfide ions. Oxidation of bromide to bromine may also be thought of as charging

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the bromine/bromide electrolyte since the chemical content of the bromine/bromide electrolyte changes in the same manner as when the RFC is in its charging cycle.

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The oxidation of sulfide to sulfur rebalances the electrolytes by oxidising the equivalent amount of sulfide which would ordinarily have been oxidised by the halogen which was reduced by reaction with migrating sulfide ions. Oxidation of polysulfide to sulfur may also be thought of as discharging the sulfide/polysulfide electrolyte since the chemical content of the sulfide/polysulfide electrolyte changes in the same manner as in the RFC when it is in its discharging cycle.

In order that rebalancing of the electrolytes may occur, it is essential that during the rebalancing process the electrolyte circulating through the $-^{\mathrm{ve}}$ chamber of the auxiliary cell should be free from polysulfide and free from bromine. The reason for this is that these chemical species are more readily reduced than water. If electrolyte 1 is circulated through the $+^{ve}$ chamber of the auxiliary cell and the electrolyte circulating through the -ve chamber of the auxiliary cell contains polysulfide, then the reaction which will occur in the -ve chamber will be reduction of polysulfide to sulfide rather than reduction of water to hydrogen and hydroxide ions. This would result in no net change in the oxidation state of the sulfur species present in the system. If reduction of water is to occur in the presence of polysulfide the $-^{\mathrm{ve}}$ electrode in the $-^{\mathrm{ve}}$ chamber must be specially constructed to starve it of polysulfide. Similarly, if electrolyte 2 is circulated through the +ve chamber of the auxiliary cell and the electrolyte circulating through the $-^{ve}$ chamber of the auxiliary cell contains

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chamber will be reduction of bromine to bromide rather than reduction of water to hydrogen and hydroxide ions. This would result in no net change in the oxidation state of the bromine species present in the system. If reduction of water is to occur in the presence of bromine the -ve electrode in the -ve chamber must be specially constructed to starve it of bromine. Inclusion of such specially constructed

10 electrodes is clearly undesirable from an economic and system maintenance viewpoint.

The rebalancing process may be applied continuously to the RFC wherein a sidestream of the bromine/bromide or sulfide/polysulfide electrolyte drawn from the mainstream is diverted through apparatus suitable for carrying out the rebalancing process. The rebalancing process may also be applied as a batch process wherein the fraction of the bromine/bromide or sulfide/polysulfide electrolyte which is removed from the RFC is treated in separate apparatus suitable for carrying out the rebalancing process before being returned to the RFC.

It will be understood by those skilled in the art that a number of reduction half-cell reactions may be used to counter the oxidation of the halide or sulfide. However, in the present invention, the other half-cell reaction under alkaline conditions involves the reduction of water to hydrogen and hydroxide ions according to the half-cell reaction shown in Equation 8 below:

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$$2H_2O + 2e^- \Rightarrow H_2 + 2OH^-$$
 Equation 8

Thus the rebalancing process may be represented by the reactions shown in Equations 9 and 10 below:

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 $2Br^{-} + 2H_{2}O \Rightarrow Br_{2} + H_{2} + 2OH^{-}$ Equation 9

 $S^{2-} + 2H_2O \Rightarrow S + H_2 + 2OH^-$ Equation 10

5 Similarly in an acidic medium the half cell reaction comprises

 $2H^+ + 2e^- \rightleftharpoons H_2$ Equation 11

10 It will be appreciated that, although—the process of oxidising the bromine/bromide or sulfide/polysulfide electrolyte can be used to rebalance the electrolytes, there is still a net loss of active sulfur species from the cell. This is because the sulfide and polysulfide ions which cross to the bromine electrolyte and are oxidised to sulfate ions are not recovered. Thus, in a preferred embodiment of the present invention, the process additionally comprises adding elemental sulfur or a sulfide salt to the sulfide/polysulfide electrolyte in an amount such as

sulfide/polysulfide electrolyte in an amount such as to restore the initial concentration of active sulfur species.

In carrying out the process of the present invention
the electrolyte circulating through the -ve chamber of
the auxiliary cell may be water. In this instance the
electrolyte will generally circulate in a closed
system and there will be no change of pH of the
bromine/bromide or sulfide/polysulfide electrolyte.

In an alternative manner of carrying out the process of the present invention the electrolyte circulating through the $-^{\rm ve}$ chamber of the auxiliary cell is a fraction of electrolyte 1 or 2 which has been made free of polysulfide or bromine by electrochemical

free of polysulfide or bromine by electrochemical reduction thereof. This may be achieved by recirculating electrolyte.1 or 2 through the -ve

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chamber of the auxiliary cell until all of the polysulfide or bromine has been reduced. The electrolyte circulating through the $-^{\text{ve}}$ chamber of the auxiliary cell may then be returned to the main stream of electrolyte 1 or 2.

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Alternatively, the electrochemical reduction of polysulfide or bromine which may be present in electrolyte 1 or 2 respectively occurs within the -ve chamber of a second auxiliary cell which comprises a +ve chamber containing an inert +ve electrode and a -ve chamber containing an inert -ve electrode, the chambers being separated from one another by a cation exchange membrane, the electrolyte circulating through the +ve chamber being a fraction of electrolyte 1 or electrolyte 2. This may be achieved by recirculating electrolyte 1 or 2 through the -ve chamber of the second auxiliary cell until all of the polysulfide or bromine has been reduced. The electrolyte circulating through the -ve chamber of the auxiliary cell may then be returned to the main stream of electrolyte 1 or 2.

Another reason why the reduction of any bromine which may be present in electrolyte 2 is important is because, as described in WO-A-00/03448, carrying out the RFC process of the present invention results in the production of sulfate ions in the bromine/bromide electrolyte as described above with reference to Equation 7. The removal of sulfate ions from the electrolyte can only be carried out by the process as described in WO-A-00/03448 in the absence of free bromine which otherwise interferes with the process. Thus, in a preferred embodiment, the electrolyte circulating through the -ve chamber of the auxiliary cell during rebalancing is a fraction of electrolyte 2 and that fraction is subsequently treated to remove sulfate ions contained therein.

In carrying out the process of the present invention elemental sulfur and/or a sulfide salt may be added to the sulfide/polysulfide electrolyte in an amount sufficient to restore the initial concentration of sulfur species.

The present invention also provides for the use, in a process for energy storage and/or power delivery comprising:

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- maintaining and circulating electrolyte flows in 10 a fully liquid system in which the active constituents are fully soluble in a single cell or in an array of repeating cell structures, each cell with a positive $(+^{ve})$ chamber containing an inert $+^{ve}$ electrode and a negative $(-^{ve})$ chamber 15 containing an inert -ve electrode, the chambers being separated from one another by a cation exchange membrane, the electrolyte circulating in the $-^{\mathrm{ve}}$ chamber of each cell during power 20 delivery containing a sulfide (electrolyte 1), and the electrolyte circulating in the $+^{ve}$ chamber during power delivery containing bromine (electrolyte 2),
- (ii) restoring or replenishing the electrolytes in the

 +ve and -ve chambers by circulating the
 electrolyte from each chamber to storage means
 comprising a volume of electrolyte greater than
 the cell volume for extended delivery of power
 over a longer discharge cycle than the cell
 volume alone would permit,
- of a process comprising:

 circulating a fraction of electrolyte 1 or

 electrolyte 2 through the +ve chamber of an

 auxiliary cell, said auxiliary cell comprising a

 +ve chamber containing an inert +ve electrode and

 a -ve chamber containing an inert -ve electrode,

 the chambers being separated from one another by

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a cation exchange membrane, the electrolyte circulating through the -ve chamber of the auxiliary cell containing water and being free from polysulfide and free from bromine during rebalancing, the auxiliary cell operating so as to oxidise sulfide ions to polysulfide or bromide ions to bromine in the +ve chamber and so as to reduce water to hydrogen and hydroxide ions in the -ve chamber,

10 for the purpose of rebalancing electrolytes 1 and 2.

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The present invention also includes within its scope apparatus for carrying out a process as described above comprising:

- 15 (i) a single cell or an array of repeating cell structures, each cell comprising; a +ve chamber containing an inert +ve electrode and a -ve chamber containing an inert -ve electrode the chambers being separated from one another by an ion exchange membrane, an electrolyte circulating in the -ve chamber of each cell which contains a sulfide during power delivery (electrolyte 1), and an electrolyte circulating in the +ve chamber which contains bromine during power delivery (electrolyte 2),
 - (ii) storage and circulation means for each electrolyte for restoring or replenishing the electrolytes in the $+^{ve}$ and $-^{ve}$ chambers,
- (iii) means for rebalancing the electrolytes

 comprising an auxiliary cell which comprises a

 +ve chamber containing an inert +ve electrode and
 a -ve chamber containing an inert -ve electrode
 the chambers being separated from one another by
 a cation exchange membrane, means for circulating
 a fraction of electrolyte 1 or 2 through the +ve
 chamber of the auxiliary cell, an electrolyte
 containing water and being free from polysulfide



and free from bromine during rebalancing and means for circulating said electrolyte through the $-^{\text{ve}}$ chamber of the auxiliary cell.

- 5 The present invention will be further described with reference to the accompanying drawings in which:
 - Fig 1A is a schematic view of a basic electrochemical reduction-oxidation cell in which a
- sulfide/polysulfide reaction is carried out in one half of the cell and a bromine/bromide reaction is carried out in the other half of the cell;
- Fig 1B is a diagram of cell arrays using the system of 15 Fig 1A;
 - Fig 2 is a block diagram of a fluid flow system using the cell of Fig 1A;
- Fig 3 is a flow diagram of an apparatus for carrying out a preferred embodiment of the process of the present invention.
- Fig 4 is a flow diagram of an apparatus for carrying out a preferred embodiment of the process of the present invention.
- Fig 5 is a schematic diagram of an apparatus for carrying out a further preferred embodiment of the process of the present invention, including the removal of sulfate.
- Fig 6 is a flow diagram of an apparatus for carrying out a preferred embodiment of the process of the present invention, including the removal of sulfate.
 - Fig 7 is a graph of voltage versus time for a selected

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number of cycles of a RFC which does not incorporate a rebalancing process in accordance with the present invention.

- Fig 8 is a graph of voltage versus time for a selected number of cycles of a RFC which does incorporate a rebalancing process in accordance with the present invention.
- Fig 1A shows a cell 10 with a positive (+ve) electrode 12 and a negative (-ve) electrode 14 and a cation exchange membrane 16 which may be formed from a fluorocarbon polymer with sulfonic acid functional groups to provide charge carriers. The membrane 16
- acts to separate the $+^{ve}$ and $-^{ve}$ sides of the cell 10 and is selected to minimize migration of bromine from the $+^{ve}$ side to the $-^{ve}$ side and to minimize migration of sulfide and polysulfide ions from the $-^{ve}$ side to the $+^{ve}$ side. An aqueous solution 22 of NaBr is
- provided in a chamber 22C formed between the + $^{\rm ve}$ electrode 12 and the membrane 16 and an aqueous solution 24 of Na₂S_x (where x may be from 2 to 5) is provided in a chamber 24C formed between the - $^{\rm ve}$ electrode 14 and the membrane 16. A K₂S_x solution,
- which is more soluble and more expensive than the Na_2S_{κ} solutions, is used in another embodiment.

When the cell is in the discharged state, a solution of NaBr of up to 6.0 molar concentration exists in the chamber 22C of the cell and a solution of Na_2S_x at 0.5 to 1.5 molar, exists in chamber 24C of the cell. Higher molarity is possible with K_2S_x .

As the cell is charged, Na⁺ ions are transported through the cation membrane 16, as shown in Fig 1A, from the +^{ve} to the -^{ve} side of the cell. Free bromine is produced via oxidation of the bromide ions at the

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 $+^{ve}$ electrode and dissolves as a tribromide or pentabromide ion. Sulfur is reduced at the $-^{ve}$ electrode and the pentasulfide, Na_2S_x , salt eventually becomes the monosulfide as the charging proceeds to completion. At the $+^{ve}$ side the following reaction occurs,

$$2Br^- \Rightarrow Br_2 + 2e^-$$

10 and at the -ve side the following reaction occurs,

$$S + 2e^- \Rightarrow S^{2-}$$
.

The membrane separates the two electrolytes and prevents bulk mixing and also retards the migration of sulfide and polysulfide ions from the $-^{\text{ve}}$ side to the $+^{\text{ve}}$ side, and the migration of Br^- and Br_2 from the $+^{\text{ve}}$ to the $-^{\text{ve}}$ side. Diffusion of the sulfide and polysulfide ions across the membrane results in the electrolytes becoming unbalanced as described earlier.

When providing power, the cell is discharging. During this action, reversible reactions occur at the two electrodes. At the $+^{ve}$ side electrode 12, bromine is reduced to Br $^-$, and at the $-^{ve}$ electrode, the S $^{2-}$ ion is oxidized to molecular S. The electrons produced at the $-^{ve}$ electrode form the current through a load. The chemical reaction at the $+^{ve}$ electrode produces 1.06 to 1.09 volts and the chemical reaction at the $-^{ve}$ electrode produces 0.48 to 0.52 volts. The combined chemical reactions produce an open circuit voltage of 1.54 to 1.61 volts per cell.

The present system is an anionically active
35 electrochemical system. Therefore, the cation which is
associated with them essentially takes no part in the
energy producing process. Hence, a cation of

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"convenience" is chosen. Sodium or potassium are preferred choices. Sodium and potassium compounds are plentiful, they are inexpensive and have high water solubilities. Lithium and ammonium salts are also possibilities, but at higher costs.

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Fig 1B shows an array 20 of multiple cells connected in electrical series and fluid parallel. Multiple midelectrodes 13 (each one having a $+^{ve}$ electrode side 12A and $-^{ve}$ electrode side 14A) and end electrodes 12E $(+^{ve})$ and 14E $(-^{ve})$ are spaced out from each other by membranes 16 and screen or mesh spacers (22D, 24D) in all the cell chambers 22C, 24C, (portions of two of which 22D, 24D are shown by way of example) to form end cells C_{E1} and C_{E2} and an array of mid cells C_{M} (typically 10-20; but note much smaller and much higher numbers of cells can be accommodated). The end electrodes 12E $(+^{ve})$ and 14E $(-^{ve})$ have internal conductors 12F and 14F (typically copper screens) encapsulated therein and leading to external terminals 12G, 14G which are connected to external loads (e.g. to motor(s) via a control circuit (CONT), the motor(s) may be used to drive a vehicle) or power sources (e.g. utility power grid when used as a load-levelling device).

Fig 2 shows a free flow system, a power generation/storage system utilizing one or more of the batteries or cell array formats 20. Each cell 20 receives electrolyte through pumps 26 and 28 for the NaBr and Na₂S_x solutions (22 and 24, respectively). The electrolytes 22 and 24 are stored in containers 32 and 34. The tanks 32, 34 can be replaced with freshly charged electrolyte by substituting tanks containing fresh electrolyte and/or refilling them from charged supply sources via lines 32R, 34R with corresponding lines (not shown) provided for draining spent

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(discharged) reagent. The electrolytes 22 and 24 are pumped from tanks 32 and 34, respectively, into the respective chambers 22C and 24C by means of pumps 26 and 28.

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Fig 3 shows a free flow system in which an array of cells 20 are supplied with bromine/bromide and sulfide/polysulfide electrolyte from storage tanks 41 and 42 via lines 43 and 44. Bromine/bromide electrolyte may be removed from storage tank 41 via line 50 which transfers it to an external electrochemical cell 51 wherein the bromide is oxidised to bromine as a half-cell reaction in an electrochemical process. The other half-cell reaction involves the reduction of water to hydrogen and hydroxide ions. An aqueous electrolyte is stored in tank 52 and transported to the electrochemical cell 51 via line 53. The reduced electrolyte is passed via line 54 to tank 55 where hydrogen gas which is generated by the electrochemical reaction may be vented from the system. The electrolyte returns via

Fig 4 shows a particularly preferred variation of the
free flow system illustrated in Fig 3. In this
embodiment the aqueous electrolyte which is reduced in
the external electrochemical cell 51 is also
bromine/bromide electrolyte which has been removed
from storage tank 41 to tank 52 via line 57. In this
case, the reduction reaction will initially involve
reduction of any residual bromine to bromide and will
subsequently involve reduction of water to hydrogen
and hydroxide ions. The reduced electrolyte may be
subsequently returned to the storage tank 41 along
line 58.

line 56 to storage tank 52.

Referring to Fig 5, a schematic flow diagram is shown

of the manner in which the electrolyte may be treated. A storage tank 41 contains the aqueous bromine/bromide electrolyte which may be circulated around the main RFC system (not shown). The first treatment of a first stream of the electrolyte from tank 41 is to 5 remove free bromine by treatment in an appropriate bromine reduction module 61 which comprises an auxiliary cell. The electrolyte is circulated via module 61 and an intermediate storage tank 62 until 10 the bromine present in the electrolyte is reduced to bromide. When the reduction is complete the electrolyte is passed to storage tank 63. A second stream of the electrolyte from tank 41 is passed to an electrochemical cell module 64 where the bromide is 15 oxidised to bromine as a half-cell reaction in an electrochemical process. The other half-cell reaction involves the reduction of water to hydrogen and hydroxyl ions using as the electrolyte for the hydroxyl ion production the electrolyte from storage 20 tank 63. The stream of electrolyte passing through module 64 in which bromide has been oxidised to bromide is returned to the storage tank 41. stream of electrolyte used in the complementary halfcell reaction may be passed to a further tank 65 where 25 it is then subjected to a sulfate removal treatment according to the teaching of WO-A-00/03448 in module The stream of electrolyte from which the sulfate has been removed is then returned to the original storage tank 41. Storage tank 63 is provided with 30 appropriate means to vent hydrogen produced in the water reduction reaction to a hydrogen stack along line 67.

Fig 6 shows an alternative system for use in accordance with the present invention which includes a sulfate crystallization unit as described in WO-A-00/03448. In this system a portion of bromide/bromine

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electrolyte contaminated with sulfate ions is drawn from the main system 80 via line 81 and held in a receiving tank 82. This electrolyte is then circulated via lines 83 and 84 through the $-^{\text{ve}}$ chamber 85 of an auxiliary cell 86 until substantially all of 5 the bromine present in the electrolyte has been reduced to bromide ions. The voltage applied across cell 86 is limited to ensure that reduction of water does not occur. When the current density has run down (thus indicating that the conversion of bromine to 10 bromide is substantially complete), the voltage applied to the cell 86 is increased to a value sufficient to cause reduction of the water present in the electrolyte so as to generate H_2 gas and OH^- ions. The electrolyte circulating through the $+^{ve}$ chamber of 15 the external auxiliary cell is either sulfide/polysulfide or bromine/bromide electrolyte taken from the main system. The oxidation of one or the other of these electrolytes rebalances the system. 20 The removal of water from the electrolyte is advantageous because it further increases the concentration of bromide ions thereby reducing the sulfate solubility and increasing the yield of sulfate on crystallisation. A tap 87 is provided to draw off When sufficient rebalancing has occurred, the 25 electrolyte circulating through the $-^{ve}$ chamber 85 is passed from the receiving tank 82 to the crystalliser 88 via line 89. This electrolyte is then passed via line 90 through a filter 91 to remove the sulfate 30 crystals and then it may be returned to the main system via line 92. The embodiment shown in Fig 6 shows an electrolyte being drawn from the main system via line 93, passing through the $+^{ve}$ chamber 94 of the auxiliary cell and returning the main system via line 35 95.

The present invention will now be further described by

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reference to the following examples.

Comparative Example 1

A regenerative fuel cell of the type described above having sulfide/polysulfide and bromine/bromide electrolytes was set up. The cell had the following specifications:

electrode material: polyethylene impregnated with

10 activated carbon

electrode area: 2000cm² current density: 80mA/cm²

electrolyte volume: 91 per electrolyte

cycle time: 6hours (i.e. 3hours charge

and 3hours discharge)

flow rate: 1000ml/min membrane material: Nafion 115™

The cell was operated over 18 cycles (108 hours) and the cell voltage was monitored throughout this period. The results are shown in Fig 7. It can be seen that after a limited number of cycles (about 7) the cell voltage limits early on the discharge cycle due to the lack of bromine. This problem gets worse as the number of cycles increases. It can be clearly seen on the graph that after about 12 cycles (72 hours) the cell fails to maintain a good voltage performance over the whole of the 3 hour discharge cycle.

30 Example 1

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An identical RFC to that used in Comparative Example 1 was set up. This time the electrolytes were continuously rebalanced by oxidation of a sidestream of the bromide/bromine electrolyte drawn from the mainstream. Oxidation occurred in one half of an external electrochemical cell wherein the electrolyte

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undergoing reduction in the other half of the cell was dilute aqueous sodium hydroxide. The external electrochemical cell used was an MP cell from Electrocell AB having the following specifications:

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anode material: platinum cathode material: nickel electrode area: 100cm² current density: 13mA/cm²

10 flow rate:

270ml/min

membrane material: Nafion 350™

Fig. 3 shows a schematic representation of the apparatus used in the present example. The cell was operated over at least 92 cycles (552 hours) and the cell voltage was monitored throughout this period. The results from the period from 400 to 550 hours are shown in Fig 8. It can be seen that even after 91 cycles (546 hours) the cell voltage does not limit early on the discharge cycle as occurred in the unbalanced cell. The cell retains a good voltage performance over the whole of the 3 hour discharge cycle.

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CLAIMS:

- 1. An electrochemical process for energy storage and/or power delivery comprising:
- maintaining and circulating electrolyte flows in 5 (i) a fully liquid system in which the active constituents are fully soluble in a single cell or in an array of repeating cell structures, each cell with a positive $(+^{ve})$ chamber containing an 10 inert $+^{ve}$ electrode and a negative $(-^{ve})$ chamber containing an inert - electrode, the chambers being separated from one another by a cation exchange membrane, the electrolyte circulating in the $-^{\mathrm{ve}}$ chamber of each cell during power 15 delivery containing a sulfide (electrolyte 1), and the electrolyte circulating in the $+^{ve}$ chamber during power delivery containing bromine (electrolyte 2),
- (ii) restoring or replenishing the electrolytes in the

 +ve and -ve chambers by circulating the
 electrolyte from each chamber to storage means
 comprising a volume of electrolyte greater than
 the cell volume for extended delivery of power
 over a longer discharge cycle than the cell
 volume alone would permit, and
- (iii) rebalancing the electrolytes by circulating a fraction of electrolyte 1 or electrolyte 2 through the +ve chamber of an auxiliary cell, said auxiliary cell comprising a +ve chamber containing an inert +ve electrode and a -ve chamber containing an inert -ve electrode, the chambers being separated from one another by a cation exchange membrane, the electrolyte circulating through the -ve chamber of the auxiliary cell containing water and being free from polysulfide and free from bromine during rebalancing, the auxiliary cell operating so as

to oxidise sulfide ions to sulfur or bromide ions to bromine in the $+^{\text{ve}}$ chamber and so as to reduce water to hydrogen and hydroxide ions in the $-^{\text{ve}}$ chamber.

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- 2. A process as claimed in claim 1 wherein the electrolyte circulating through the -ve chamber of the auxiliary cell during rebalancing is a fraction of electrolyte 1 or electrolyte 2 which has been made free of polysulfide or bromine by electrochemical reduction thereof.
- 3. A process as claimed in claim 2 wherein the electrochemical reduction of polysulfide or bromine is effected by recirculating the fraction of electrolyte 1 or 2 through the -ve chamber of the auxiliary cell until all of the polysulfide or bromine has been reduced.
- 4. A process as claimed in claim 2 wherein the electrochemical reduction of polysulfide or bromine occurs within the -ve chamber of a second auxiliary cell which comprises a +ve chamber containing an inert +ve electrode and a -ve chamber containing an inert -ve electrode, the chambers being separated from one another by a cation exchange membrane, the electrolyte circulating through the +ve chamber being a fraction
- 30 5. A process as claimed in claim 4 wherein the electrochemical reduction of polysulfide or bromine is effected by recirculating the fraction of electrolyte 1 or 2 through the -ve chamber of the second auxiliary cell until all of the polysulfide or bromine has been reduced.

of electrolyte 1 or electrolyte 2.

6. A process as claimed in any one of claims 3 to 5

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wherein the electrolyte circulating through the $-^{\text{ve}}$ chamber of the auxiliary cell during rebalancing is a fraction of electrolyte 2 and wherein that fraction is subsequently treated to remove sulfate ions contained therein.

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- 7. A process as claimed in claim 6 wherein said sulfate ions are removed by crystallisation of a sulfate salt from the fraction of electrolyte 2.
- 8. A process as claimed in any one of claims 2 to 7 wherein the fraction of electrolyte 1 or 2 which is circulated through the -ve chamber of the auxiliary cell is returned to the main stream of electrolyte 1 or 2 respectively.
 - 9. A process as claimed in any one of the preceding claims which additionally comprises adding elemental sulfur and/or a sulfide salt to electrolyte 1 in an amount sufficient to restore the initial concentration of sulfur species.
 - 10. Use, in a process for energy storage and/or power delivery comprising:
- 25 maintaining and circulating electrolyte flows in a fully liquid system in which the active constituents are fully soluble in a single cell or in an array of repeating cell structures, each cell with a positive $(+^{ve})$ chamber containing an inert $+^{ve}$ electrode and a negative $(-^{ve})$ chamber 30 containing an inert $-^{ve}$ electrode, the chambers being separated from one another by a cation exchange membrane, the electrolyte circulating in the $\ensuremath{^{-\mathrm{ve}}}$ chamber of each cell during power delivery containing a sulfide (electrolyte 1), 35 and the electrolyte circulating in the $+^{ve}$ chamber during power delivery containing bromine

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(electrolyte 2),

- (ii) restoring or replenishing the electrolytes in the +ve and -ve chambers by circulating the electrolyte from each chamber to storage means comprising a volume of electrolyte greater than the cell volume for extended delivery of power over a longer discharge cycle than the cell volume alone would permit,
- of a process comprising:

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- circulating a fraction of electrolyte 1 or 10 electrolyte 2 through the $+^{ve}$ chamber of an auxiliary cell, said auxiliary cell comprising a $+^{ve}$ chamber containing an inert $+^{ve}$ electrode and a -ve chamber containing an inert -ve electrode, 15 the chambers being separated from one another by a cation exchange membrane, the electrolyte circulating through the -ve chamber of the auxiliary cell containing water and being free from polysulfide and free from bromine during 20 rebalancing, the auxiliary cell operating so as to oxidise sulfide ions to polysulfide or bromide ions to bromine in the +ve chamber and so as to reduce water to hydrogen and hydroxide ions in the -ve chamber,
- 25 for the purpose of rebalancing electrolytes 1 and 2.
 - 11. An electrochemical apparatus for energy storage and/or power delivery comprising:
- (i) a single cell or an array of repeating cell

 structures, each cell comprising; a +ve chamber
 containing an inert +ve electrode and a -ve
 chamber containing an inert -ve electrode the
 chambers being separated from one another by an
 ion exchange membrane, an electrolyte circulating
 in the -ve chamber of each cell which contains a
 sulfide during power delivery (electrolyte 1),
 and an electrolyte circulating in the +ve chamber

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which contains bromine during power delivery (electrolyte 2),

- (ii) storage and circulation means for each electrolyte for restoring or replenishing the electrolytes in the $+^{\text{ve}}$ and $-^{\text{ve}}$ chambers,
- (iii) means for rebalancing the electrolytes
 comprising an auxiliary cell which comprises a
 +ve chamber containing an inert +ve electrode and
 a -ve chamber containing an inert -ve electrode
 the chambers being separated from one another by
 a cation exchange membrane, means for circulating
 a fraction of electrolyte 1 or 2 through the +ve
 chamber of the auxiliary cell, an electrolyte
 containing water and being free from polysulfide
 and free from bromine during rebalancing and
 means for circulating said electrolyte through
 the -ve chamber of the auxiliary cell.
- 12. Apparatus as claimed in claim 11 wherein the
 means for circulating an electrolyte through the -ve
 chamber of the auxiliary cell comprises means for
 circulating a fraction of electrolyte 1 or 2 through
 the -ve chamber of the auxiliary cell.
- 13. Apparatus as claimed in claim 11 wherein the means for circulating an electrolyte through the -ve chamber of the auxiliary cell comprises a storage tank into which a fraction of electrolyte 1 or 2 may be transferred and means for re-circulating the fraction of electrolyte 1 or 2 between the -ve chamber of the auxiliary cell and said storage tank.
- 14. Apparatus as claimed in claim 12 or claim 13 which additionally comprises a second auxiliary cell which comprises a +ve chamber containing an inert +ve electrode and a -ve chamber containing an inert -ve electrode, the chambers being separated from one

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another by a cation exchange membrane, means for circulating a fraction of electrolyte 1 or 2 through the $+^{ve}$ chamber and means for circulating a fraction of electrolyte 1 or 2 through the $-^{ve}$ chamber.

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- 15. Apparatus as claimed in claim 14 wherein the means for circulating an electrolyte through the -ve chamber of the second auxiliary cell comprises a storage tank into which a fraction of electrolyte 1 or 2 may be transferred and means for re-circulating the fraction of electrolyte 1 or 2 between the -ve chamber of the second auxiliary cell and said storage tank.
- 16. Apparatus as claimed in any one of claims 12 to 15 wherein the electrolyte circulated through the -ve chamber of the auxiliary cell is electrolyte 2, additionally comprising means for removing sulfate ions from the fraction of electrolyte 2 after circulation through the -ve chamber of the auxiliary cell.
 - 17. Apparatus as claimed in claim 16 wherein the means for removing sulfate ions from electrolyte 2 comprises a crystalliser.

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- 18. Apparatus as claimed in any one of claims 11 to 17 additionally comprising means for passing the fraction of electrolyte 1 or 2 which is circulated through the $-^{ve}$ chamber of the auxiliary cell back to the main stream of electrolyte 1 or 2 respectively.
- 19. Use in an electrochemical apparatus for energy storage and/or power delivery comprising:
- (i) a single cell or an array of repeating cell

 structures, each cell comprising; a +ve chamber containing an inert +ve electrode and a -ve chamber containing an inert -ve electrode the

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chambers being separated from one another by an ion exchange membrane, an electrolyte circulating in the $-^{\text{ve}}$ chamber of each cell which contains a sulfide during power delivery (electrolyte 1), and an electrolyte circulating in the $+^{\text{ve}}$ chamber which contains bromine during power delivery (electrolyte 2), and

(ii) storage and circulation means for each electrolyte for restoring or replenishing the electrolytes in the $+^{ve}$ and $-^{ve}$ chambers,

of

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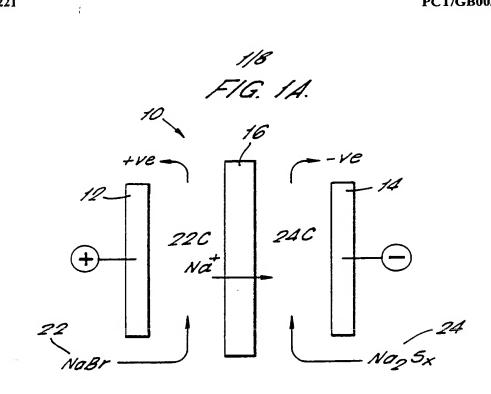
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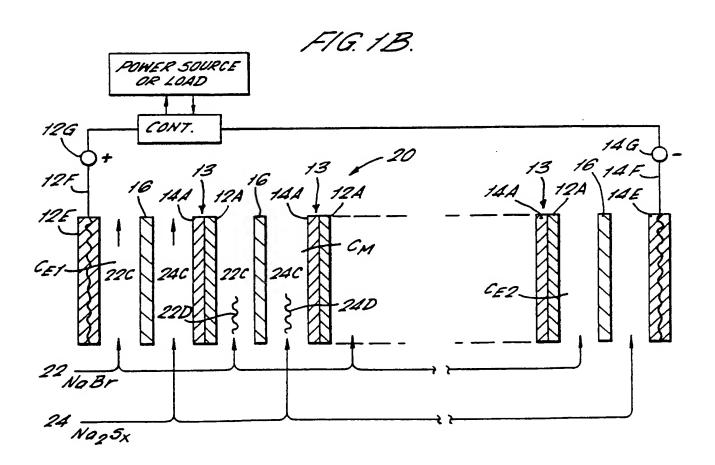
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an auxiliary cell which comprises; a $+^{ve}$ chamber containing an inert $+^{ve}$ electrode and a $-^{ve}$ chamber containing an inert $-^{ve}$ electrode the chambers being separated from one another by a cation exchange membrane, means for passing a fraction of electrolyte 1 or 2 through the $+^{ve}$ chamber of the auxiliary cell, an electrolyte containing water and being free from polysulfide and free from bromine during rebalancing and means for circulating said electrolyte through the $-^{ve}$ chamber of the auxiliary cell

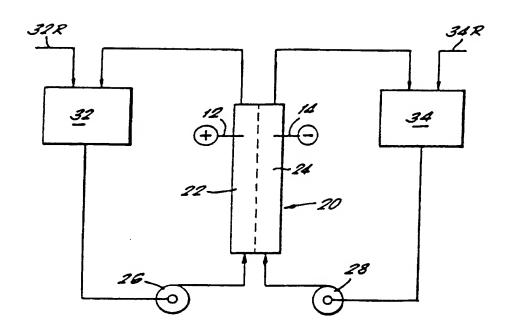
for the purpose of rebalancing electrolytes 1 and 2.

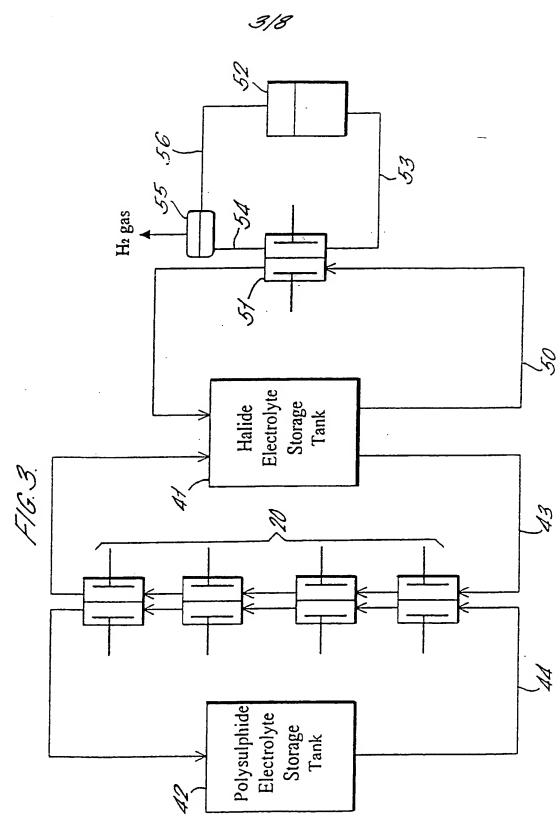




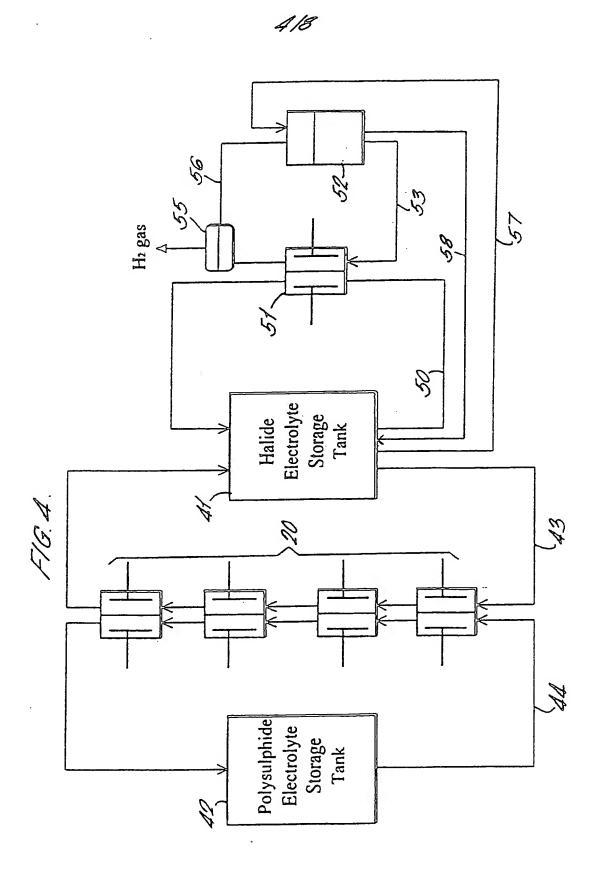
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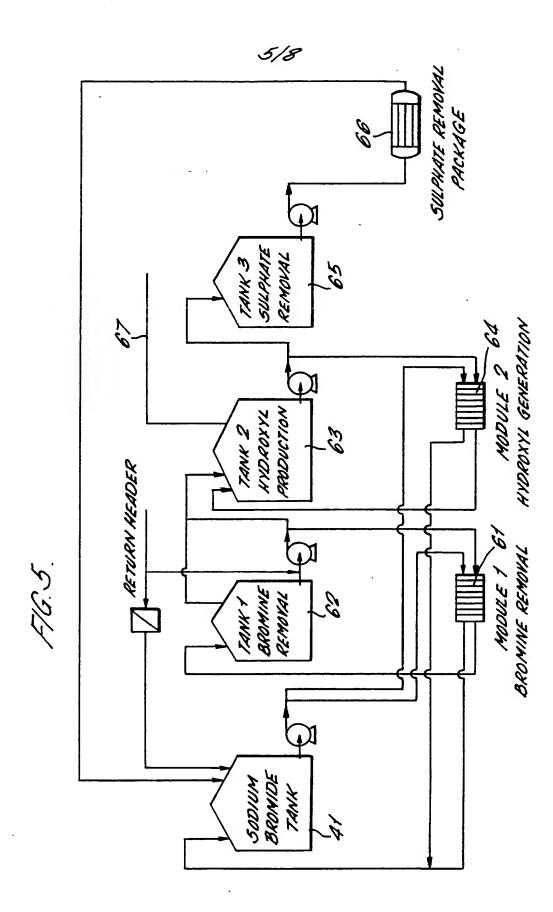
FIG. 2.



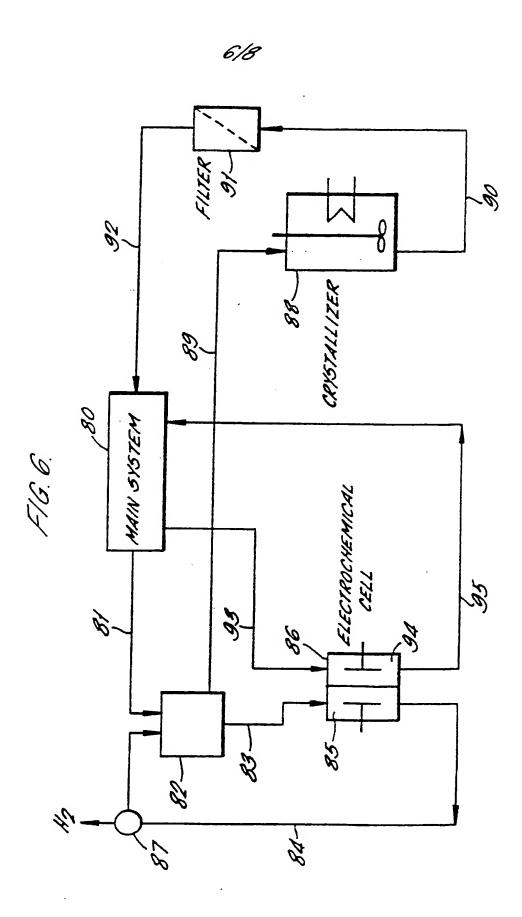






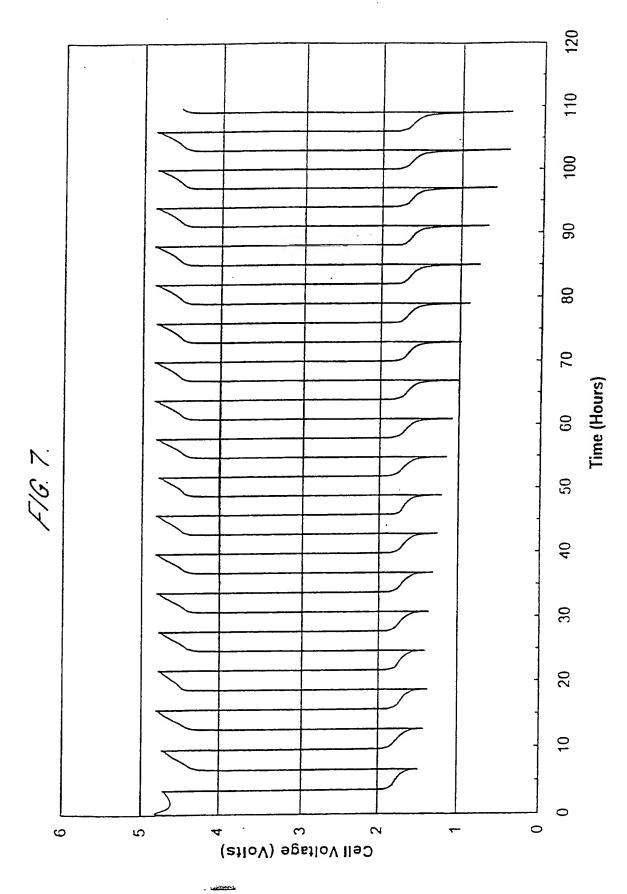


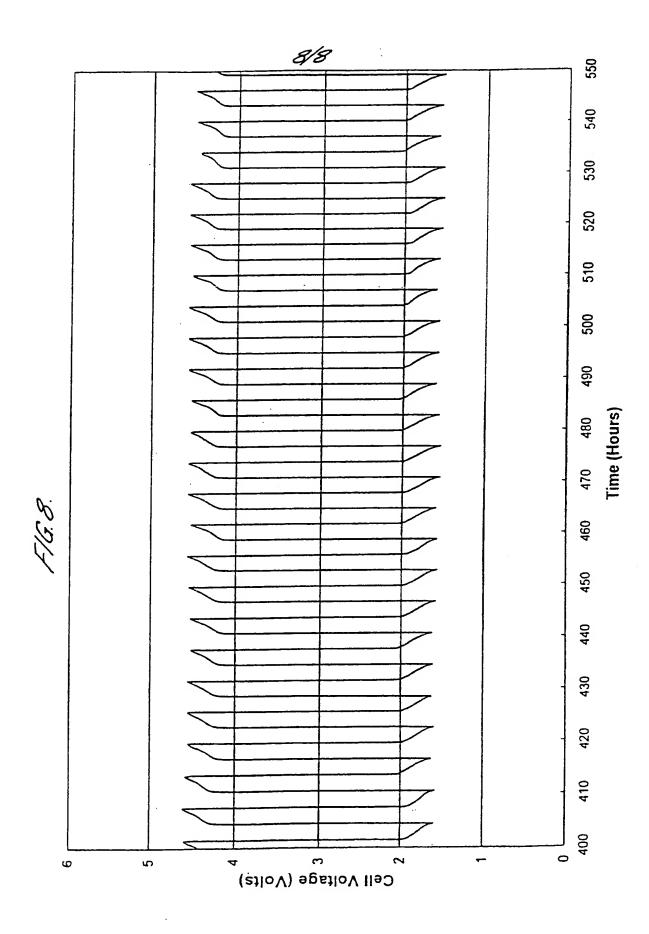
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A. CL	ASSIFICA	TION OF	SUBJECT	MAT	
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According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols) IPC 7 H01M

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

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Special categories of cited documents: "A" document defining the general state of the art which is not considered to be of particular relevance "E" earlier document but published on or after the international filing date "L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified) "O" document referring to an oral disclosure, use, exhibition or other means "P" document published prior to the international filing date but later than the priority date claimed	"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention "X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone "Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art. "&" document member of the same patent family
Date of the actual completion of the international search	Date of mailing of the international search report
17 October 2000	24/10/2000
Name and mailing address of the ISA European Patent Office, P.B. 5818 Patentlaan 2	Authorized officer
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WO	9409526	Α		ZA	9307284	Α	30-03-1995
WO	9409525	A	28-04-1994	AT	152860	T	15-05-1997
				AT	162338	T	15-01-1998
		-		ΑU	672049	В	19-09-1996
				AU	5153793	Α	09-05-1994
				ΑU	676691	В	20-03-1997
				AU	5153893	Α	09-05-1994
				CN	1087752	A,B	08-06-1994
				DE	69310529	D	12-06-1997
				DE	69310529	T	06-11-1997
				DE	69316387	D	19-02-1998
				DE	69316387	T	28-05-1998
				DK	664931	T	08-12-1997
				DK	664932	T	14-09-1998
				EG	20201	Α	30-10-1997
				EP	0664931	Α	02-08-1995
				ΕP	0664932	Α	02-08-1995
				ES	2104179	T	01-10-1997
				ES	2111774	T	16-03-1998
				WO	9409526	Α	28-04-1994
				GR	3024385	T	28-11-1997
				GR	3026051	T	29-05-1998
				HK	1007461	Α	09-04-1999
				IL	107237	Α	14-05-1996
				JP	8502387	T	12-03-1996
				JP	8502388	T	12-03-1996
				US	5496659	A	05-03-1996
				ZA	9307284		30-03-1995
				US 	5422197	A 	06-06-1995 
DE	3522714	Α	08-01-1987	NONE	•		

PCT	For receiving Office use only
	International Application No.
REQUEST	International Filing Date
The undersigned requests that the present international application be processed according to the Patent Cooperation Treaty.	Name of receiving Office and "PCT International Application"
	Applicant's or agent's file reference (if desired) (12 characters maximum) SJA/53278/00 6
Box No. I TITLE OF INVENTION	•
ELECTROLYTE REBALANCING SYSTEM	
Box No. II APPLICANT	
Name and address: (Family name followed by given name; for designation. The address must include postal code and name of address indicated in this Box is the applicant's State (that is, court of residence is indicated below.)  NATIONAL POWER PLC  Windmill Hill Business Park  Whitehill Way	r a legal entity, full official country. The country of the intry) of residence if no State  Telephone No.  Facsimile No.
Swindon, Wiltshire SN5 6PB United Kingdom	Teleprinter No.
State (that is, country) of nationality: United Kingdom	State (that is, country) of residence: United Kingdom
This person is applicant for the purposes of:  all designated sall designated States  all designated the Unite	nated States except ed States of America the United States of Lates of America only the Supplemental Box
Box No. III FURTHER APPLICANT(S) AND/OR (FU	RTHER) INVENTOR(S)
Name and address: (Family name followed by given name; for designation. The address must include postal code and name of address indicated in this Box is the applicant's State (that is, court of residence is indicated below.) MORRISSEY; Patrick John 11 Bryong Close Hillingdon Middlesex UB8 3RB United Kingdom	This person is:  This person is:  applicant only  applicant and inventor  inventor only (If this check-box is marked, do not fill in below.)
State (that is, country) of nationality: Ireland	State (that is, country) of residence: Unbited Kingdom
This person is applicant for the purposes of:  all designated all designated the United States.	nated States except of America only the States indicated in the Supplemental Box
Further applicants and/or (further) inventors are indicated	ed on a continuation sheet.
Box No. IV AGENT OR COMMON REPRESENTATI	IVE; OR ADDRESS FOR CORRESPONDENCE
The person identified below is hereby/has been appointed to a of the applicant(s) before the competent International Authori	

BOULT WADE TENNANT

**VERULAM GARDENS** 70 GRAY'S INN ROAD

**LONDON WC1X 8BT** 

UNITED KINGDOM

Name and address: (Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country.)

Telephone No.

Facsimile No.

Teleprinter No.

+44 (0)20 7430 7500

+44 (0)20 7831 1768

Sheet No	
Continuation of Box No. III FURTHER APPLICANT(S) AND	O/OR (FURTHER) INVENTOR(S)
If none of the following sub-boxes is used, this s	sheet should not be included in the request.
Name and address: (Family name followed by given name; for a lega designation. The address must include postal code and name of country address indicated in this Box is the applicant's State (that is, country) of of residence is indicated below.) MITCHELL; Philip John 1 Compton Close Loughborough LE1 13SF United Kingdom	al entity, full official The country of the residence if no State  This person is:  applicant only  applicant and inventor  inventor only (If this check-box is marked, do not fill in below.)
	itate (that is, country) of residence: nited Kingdom
This person is applicant for the purposes of:  all designated States all designated States all designated States	the States indicated in the Supplemental Box
Name and address: (Family name followed by given name; for a lega designation. The address must include postal code and name of country, address indicated in this Box is the applicant's State (that is, country) of of residence is indicated below.)  MALE; Stewart Ernest 68 Farm Close East Grinstead West Susex RH19 3QG United Kingdoim	This person is:  The country of the residence if no State  This person is:  applicant only  Applicant and inventor  inventor only (If this check-box is marked, do not fill in below.)
	State (that is, country) of residence: nited Kingdom
This person is applicant for the purposes of:  all designated States all designated Stat	tes except of America only the United States the States indicated in the Supplemental Box
Name and address: (Family name followed by given name; for a lega designation. The address must include postal code and name of country address indicated in this Box is the applicant's State (that is, country) of the of residence is indicated below.)	This person is:  The country of the residence if no State  This person is:  applicant only  applicant and inventor  inventor only (If this check-box is marked, do not fill in below.)
State (that is, country) of nationality:	State (that is, country) of residence:
This person is applicant for the purposes of:  all designated all designated the United States	the United States the States indicated in the Supplemental Box
Name and address: (Family name followed by given name; for a lega designation. The address must include postal code and name of country address indicated in this Box is the applicant's State (that is, country) of of residence is indicated below.)	This person is:  This person is:  applicant only  applicant and inventor  inventor only (If this check-box is marked, do not fill in below.)
State (that is, country) of nationality:	tate (that is, country) of residence:
This person is applicant all designated for the purposes of:	ates except sof America only the States indicated in the Supplemental Box
Further applicants and/or (further) inventors are indicated on a	nother continuation sheet.

#### Sheet No. 3. . . . . . DESIGNATION OF STATES The following designations are hereby made under Rule 4.9(a) (mark the applicable check-boxes; at least one must be marked): Regional Patent ARIPO Patent: GH Ghana, GM Gambia, KE Kenya, LS Lesotho, MW Malawi, SD Sudan, SL Sierra Leone, SZ Swaziland, TZ United Republic of Tanzania, UG Uganda, ZW Zimbabwe, and any other State which is a Contracting State of the Harare Protocol and of the PCT Eurasian Patent: AM Armenia, AZ Azerbaijan, BY Belarus, KG Kyrgyzstan, KZ Kazakhstan, MD Republic of Moldova, RU Russian Federation, TJ Tajikistan, TM Turkmenistan, and any other State which is a Contracting State of the Eurasian Patent Convention and of the PCT European Patent: AT Austria, BE Belgium, CH and LI Switzerland and Liechtenstein, CY Cyprus, DE Germany, DK Denmark, ES Spain, FI Finland, FR France, GB United Kingdom, GR Greece, IE Ireland, IT Italy, LU Luxembourg, MC Monaco, NL Netherlands, PT Portugal, SE Sweden, and any other State which is a Contracting State of the European Patent **EP** Convention and of the PCT OAPI Patent: BF Burkina Faso, BJ Benin, CF Central African Republic, CG Congo, CI Côte d'Ivoire, CM Cameroon, GA Gabon, GN Guinea, GW Guinea-Bissau, ML Mali, MR Mauritania, NE Niger, SN Senegal, TD Chad, TG Togo, and any other State which is a member State of OAPI and a Contracting State of the PCT (if other kind of protection or treatment desired, specify on dotted line) National Patent (if other kind of protection or treatment desired, specify on dotted line): ★ AE United Arab Emirates LR Liberia X AL Albania ...... X LT Lithuania AT Austria ..... **X** LU Luxembourg **LV** Latvia ★ AZ Azerbaijan BA Bosnia and Herzegovina ....... MD Republic of Moldova ..... BB Barbados MG Madagascar ...... BG Bulgaria ..... MK The former Yugoslav Republic of Macedonia . . . BR Brazil ..... MN Mongolia X CA Canada MW Malawi ...... CH and LI Switzerland and Liechtenstein **▼** MX Mexico NO Norway CR Costa Rica ..... $\mathbf{X}$ NZ New Zealand ..... CU Cuba ..... **▼** PL Poland ..... Z CZ Czech Republic ..... **▼** PT Portugal ..... **⋉** RO Romania X RU **DM** Dominica **⊠** SD Sudan EE Estonia ...... X SE Sweden ES Spain ..... **⊠** SG Singapore ⊠ SI **☒ GB** United Kingdom **⋉** SK X SL **▼ GD** Grenada X TJ X TR HR Croatia ..... X TZ United Republic of Tanzania HU Hungary ..... X ID Ukraine ..... Indonesia Uganda ..... IL Israel ..... **⊠** UG **⋈** IN

✓ KZ Kazakhstan
 ✓ AG Antigua & Barbuda BZ Belize DO Dominican Republic
 ✓ LC Saint Lucia
 ✓ DZ Algeria
 ✓ DZ Algeria
 ✓ MZ Mozambique
 ✓ Precautionary Designation Statement: In addition to the designations made above, the applicant also makes under Rule 4.9(b) all other

X VN

**⋈** YU

X ZA

■ UZ Uzbekistan ......

Yugoslavia .....

South Africa .....

Check-boxes reserved for designating States which have become party to the PCT after issuance of this sheet:

designations which would be permitted under the PCT except any designation(s) indicated in the Supplemental Box as being excluded from the scope of this statement. The applicant declares that those additional designations are subject to confirmation and that any designation which is not confirmed before the expiration of 15 months from the priority date is to be regarded as withdrawn by the applicant at the expiration of that time limit. (Confirmation (including fees) must reach the receiving Office within the 15-month time limit.)

Japan ......

KP Democratic People's Republic of Korea . . . .

KR Republic of Korea ......

X IS

**▼** JP

Iceland

Sheet No. PRIORITY CLAIM Further priority claims are indicated in the Supplemental Box. Box No. VI Number Where earlier application is: Filing date of earlier application of earlier application national application: regional application:* international application: (day/month/year) country regional Office receiving Office item (1) PCT/GB99/02103 GB 02/07/1999 item (2) 9928344.2 GB 30/11/1999 item (3) The receiving Office is requested to prepare and transmit to the International Bureau a certified copy of the earlier application(s) (only if the earlier application was filed with the Office which for the purposes of the present international application is the receiving Office) identified above as item(s): * Where the earlier application is an ARIPO application, it is mandatory to indicate in the Supplemental Box at least one country party to the Paris Convention for the Protection of Industrial Property for which that earlier application was filed (Rule 4.10(b)(ii)). See Supplemental Box. INTERNATIONAL SEARCHING AUTHORITY Choice of International Searching Authority (ISA) (if two or more International Searching Authorities are competent to carry out the international search, indicate the Authority chosen; the two-letter code may be used): Request to use results of earlier search; reference to that search (if an earlier search has been carried out by or requested from the International Searching Authority): Number Country (or regional Office) Date (day/month/year) ISA / Box No. VIII CHECK LIST; LANGUAGE OF FILING This international application contains This international application is accompanied by the item(s) marked below: the following number of sheets: 1. X fee calculation sheet 2. 

separate signed power of attorney description (excluding 3. copy of general power of attorney; reference number, if any: : 21 sequence listing part) 4. statement explaining lack of signature claims : 7 5. priority document(s) identified in Box No. VI as item(s): abstract : 1 6. Translation of international application into (language): drawings : 8 sequence listing part 7. 

separate indications concerning deposited microorganism or other biological material of description 8. nucleotide and/or amino acid sequence listing in computer readable form 9. dother (specify): Total number of sheets: 41 Language of filing of the Figure of the drawings which **ENGLISH** should accompany the abstract: international application: SIGNATURE OF APPLICANT OR AGENT Next to each signature, indicate the name of the person signing and the capacity in which the person signs (if such capacity is not obvious from reading the request). ALLARD: Susan Joyce **BOULT WADE TENNANT** 30th June, 2000

_		For receiving Office use only	,
1.	Date of actual receipt of the purported international application:		2. Drawings:
3.	Corrected date of actual receipt due to later but timely received papers or drawings completing the purported international application:		received:
4.	Date of timely receipt of the required corrections under PCT Article 11(2):		not received:
5.	International Searching Authority (if two or more are competent): ISA /	6. Transmittal of search copy delayed until search fee is paid.	

For International Bureau use only

Date of receipt of the record copy by the International Bureau:

# PATENT COOPERATION TREATY Miss allord (only)

411-8/5/4 A7. MADIS - 2/1/02

From the

INTERNATIONAL PRELIMINARY EXAMINING AUTHORITY

To:

BOULT WADE TENNANT Verulam Gardens 70 Gray's Inn Road London WC1X 8BT GRANDE BRETAGNE



30/06/2000

PCT

NOTIFICATION OF TRANSMITTAL OF THE INTERNATIONAL PRELIMINARY EXAMINATION REPORT

(PCT Rule 71.1)

Date of mailing (day/month/year)

04.05.2001

Applicant's or agent's file reference

SJA/53278/006

International application No. PCT/GB00/02536

International filing date (day/month/year)

Priority date (day/month/year)

IMPORTANT NOTIFICATION

02/07/1999

Applicant

NATIONAL POWER PLC et al.

- 1. The applicant is hereby notified that this International Preliminary Examining Authority transmits herewith the international preliminary examination report and its annexes, if any, established on the international application.
- 2. A copy of the report and its annexes, if any, is being transmitted to the International Bureau for communication to all the elected Offices.
- 3. Where required by any of the elected Offices, the International Bureau will prepare an English translation of the report (but not of any annexes) and will transmit such translation to those Offices.

#### 4. REMINDER

The applicant must enter the national phase before each elected Office by performing certain acts (filing translations and paying national fees) within 30 months from the priority date (or later in some Offices) (Article 39(1)) (see also the reminder sent by the International Bureau with Form PCT/IB/301).

Where a translation of the international application must be furnished to an elected Office, that translation must contain a translation of any annexes to the international preliminary examination report. It is the applicant's responsibility to prepare and furnish such translation directly to each elected Office concerned.

For further details on the applicable time limits and requirements of the elected Offices, see Volume II of the PCT Applicant's Guide.

Name and mailing address of the IPEA/

Authorized officer

9)

European Patent Office D-80298 Munich Tel. +49 89 2399 - 0 Tx: 523656 epmu d Fax: +49 89 2399 - 4465

Myers, J

Tel.+49 89 2399-8111





# **PCT**

## INTERNATIONAL PRELIMINARY EXAMINATION REPORT

(PCT Article 36 and Rule 70)

, ,	or agent's file reference	FOR FURTHER ACTION	See Notification of Transmittal of International
SJA/532	5JA/53278/006		Preliminary Examination Report (Form PCT/IPEA/416)
	al application No.	International filing date (day/mo	
PCT/GB	00/02536	30/06/2000	02/07/1999
Internation H01M8/0		or national classification and IPC	
Applicant			·
NATION	AL POWER PLC et al.		
		examination report has been preparant according to Article 36.	pared by this International Preliminary Examining Authority
2. This	REPORT consists of a tot	al of 4 sheets, including this cover	er sheet.
. t	een amended and are the	e basis for this report and/or sheet on 607 of the Administrative Instru	of the description, claims and/or drawings which have ets containing rectifications made before this Authority ructions under the PCT).
3. This	report contains indications  Basis of the report	s relating to the following items:	
II	☐ Priority		
III			, inventive step and industrial applicability
V			d to novelty, inventive step or industrial applicability;
VI	☐ Certain document	· •	•
VII	☐ Certain defects in	the international application	
VIII	☐ Certain observatio	ns on the international application	n
Date of sub	omission of the demand	Date	e of completion of this report
29/01/20	01	04.05	05.2001
	mailing address of the internal examining authority: European Patent Office D-80298 Munich	Fitzp	horized officer zpatrick, J
Tel. +49 89 2399 - 0 Tx: 523656 epmu d Fax: +49 89 2399 - 4465			enhane No. ±49.89.2399.8570



# INTERNATIONAL PRELIMINARY EXAMINATION REPORT

International application No. PCT/GB00/02536

1.	<b>Basis</b>	of the	report
----	--------------	--------	--------

	and		response to an invitation under Article 14 are referred to in this report as "originally filed" of this report since they do not contain amendments (Rules 70.16 and 70.17)):
	1-2	1	as originally filed
	Cla	ims, No.:	
	1-1	9	as originally filed
	Dra	wings, sheets:	
	1/8	-8/8	as originally filed
			·
2.			juage, all the elements marked above were available or furnished to this Authority in the international application was filed, unless otherwise indicated under this item.
	The	ese elements were a	available or furnished to this Authority in the following language: , which is:
		the language of a	translation furnished for the purposes of the international search (under Rule 23.1(b)).
		the language of pu	ublication of the international application (under Rule 48.3(b)).
		the language of a 55.2 and/or 55.3).	translation furnished for the purposes of international preliminary examination (under Rule
3.			eleotide and/or amino acid sequence disclosed in the international application, the y examination was carried out on the basis of the sequence listing:
		contained in the in	ternational application in written form.
		filed together with	the international application in computer readable form.
		furnished subsequ	ently to this Authority in written form.
		furnished subsequ	ently to this Authority in computer readable form.
			t the subsequently furnished written sequence listing does not go beyond the disclosure in pplication as filed has been furnished.
		The statement tha listing has been fu	t the information recorded in computer readable form is identical to the written sequence rnished.
4.	The	amendments have	e resulted in the cancellation of:
		the description,	pages:
		the claims,	Nos.:

1. With regard to the elements of the international application (Replacement sheets which have been furnished to

# INTERNATIONAL PRELIMINARY EXAMINATION REPORT

International application No. PCT/GB00/02536

		the drawings,	sheets:		
5.		•			ome of) the amendments had not been made, since they have beer as filed (Rule 70.2(c)):
		(Any replacement sh report.)	neet contair	ning such	amendments must be referred to under item 1 and annexed to this
6.	Add	itional observations, i	f necessar	y:	
٧.		soned statement un tions and explanatio			ith regard to novelty, inventive step or industrial applicability;
1.	Stat	ement			
	Nov	relty (N)	Yes: No:	Claims Claims	1-19
	Inve	entive step (IS)	Yes:	Claims	1-19

2. Citations and explanations see separate sheet

Industrial applicability (IA)

No:

No:

Yes:

Claims

Claims

Claims 1-19

# INTERNATIONAL PRELIMINARY Inter EXAMINATION REPORT - SEPARATE SHEET

International application No. PCT/GB00/02536

## Section V.2: Citations and Explanations

D1 = WO 94 09522 D2 = US 5 612 148

With particular respect to the specific passages cited in the International Search Report (ISR), although documents D1 and D2 respectively use external electrochemical cells for the oxidation of halide and sulfide or bisulfide, neither these nor the remaining documents of the ISR anticipate or fairly suggest the method of rebalancing the electrolytes of part (iii) of current claim 1. Moreover, none of the cell systems of the prior art would be suitable for carrying out said method. In particular, taking document D1 as closest state of the art, on the basis of it's disclosure, the **genuine apparatus** features of current main claim 11 are distinguished over that of Fig.3A in combination with Fig. 3B or 3C of D1 via:

- the cation exchange membrane of the auxiliary cell.
- the means for circulating an electrolyte through the -ve chamber of the auxiliary cell
- the provision of means for **alternatively** circulating electrolyte 1 or 2 through the +ve chamber of the auxiliary cell.

These all contribute to ensuring added versatility as well as improved electrolyte rebalancing over the prior art whilst minimising the undesirable reduction of polysulfide to sulfide and oxidation of polysulphide/sulfide to "lost" sulphate. This ensures that the requirements of Art.33(3) PCT are also fulfilled.



### PCT From the INTERNATIONAL SEARCHING AUTHORITY To: NOTIFICATION OF TRANSMITTAL OF **BOULT WADE TENNANT** THE INTERNATIONAL SEARCH REPORT Verulam Gardens OR THE DECLARATION Miss Alkard Jown Exam 2/2/01 70 Gray's Inn Road London WC1X 8BT (PCT Rule 44.1) UNITED KINGDOM : Dary. Date of mailing (day/month/year) 24/10/2000 Applicant's or agent's file reference FOR FURTHER ACTION See paragraphs 1 and 4 below SJA/53278/006 International application No. International filing date (day/month/year) 30/06/2000 PCT/GB 00/02536 Applicant NATIONAL POWER PLC et al. 1. X The applicant is hereby notified that the International Search Report has been established and is transmitted herewith. Filing of amendments and statement under Article 19: The applicant is entitled, if he so wishes, to amend the claims of the International Application (see Rule 46): The time limit for filing such amendments is normally 2 months from the date of transmittal of the International Search Report; however, for more details, see the notes on the accompanying sheet. RECEIVED Where? Directly to the International Bureau of WIPO 34, chemin des Colombettes 1211 Geneva 20, Switzerland Fascimile No.: (41-22) 740.14.35 BOULT WADE TENNANTunder For more detailed instructions, see the notes on the accompanying sheet. The applicant is hereby notified that no International Search Report will be established and that Article 17(2)(a) to that effect is transmitted herewith. With regard to the protest against payment of (an) additional fee(s) under Rule 40.2, the applicant is notified that: the protest together with the decision thereon has been transmitted to the International Bureau together with the applicant's request to forward the texts of both the protest and the decision thereon to the designated Offices. no decision has been made yet on the protest; the applicant will be notified as soon as a decision is made. 4. Further action(s): The applicant is reminded of the following: Shortly after 18 months from the priority date, the international application will be published by the International Bureau. If the applicant wishes to avoid or postpone publication, a notice of withdrawal of the international application, or of the priority claim, must reach the International Bureau as provided in Rules 90bis.1 and 90bis.3, respectively, before the completion of the technical preparations for international publication. Within 19 months from the priority date, a demand for international preliminary examination must be filed if the applicant wishes to postpone the entry into the national phase until 30 months from the priority date (in some Offices even later). Within 20 months from the priority date, the applicant must perform the prescribed acts for entry into the national phase before all designated Offices which have not been elected in the demand or in a later election within 19 months from the priority date or could not be elected because they are not bound by Chapter II.

Authorized officer

Maria Van der Hoeven

Name and mailing address of the International Searching Authority

Fax: (+31-70) 340-3016

European Patent Office, P.B. 5818 Patentlaan 2 NL-2280 HV Rijswijk Tel. (+31-70) 340-2040, Tx. 31 651 epo nl,

#### N TO FORM PCT/ISA/220

These Notes are intended to give the basic instructions concerning the filing of amendments under article 19. The Notes are based on the requirements of the Patent Cooperation Treaty, the Regulations and the Administrative Instructions under that Treaty. In case of discrepancy between these Notes and those requirements, the latter are applicable. For more detailed information, see also the PCT Applicant's Guide, a publication of WIPO.

In these Notes, "Article", "Rule", and "Section" refer to the provisions of the PCT, the PCT Regulations and the PCT Administrative Instructions respectively.

#### INSTRUCTIONS CONCERNING AMENDMENTS UNDER ARTICLE 19

The applicant has, after having received the international search report, one opportunity to amend the claims of the international application. It should however be emphasized that, since all parts of the international application (claims, description and drawings) may be amended during the international preliminary examination procedure, there is usually no need to file amendments of the claims under Article 19 except where, e.g. the applicant wants the latter to be published for the purposes of provisional protection or has another reason for amending the claims before international polication. Furthermore, it should be emphasized that provisional protection is available in some States only.

#### What parts of the international application may be amended?

Under Article 19, only the claims may be amended.

During the international phase, the claims may also be amended (or further amended) under Article 34 before the International Preliminary Examining Authority. The description and drawings may only be amended under Article 34 before the International Examining Authority.

Upon entry into the national phase, all parts of the international application may be amended under Article 28 or, where applicable, Article 41.

#### When?

Within 2 months from the date of transmittal of the international search report or 16 months from the priority date, whichever time limit expires later. It should be noted, however, that the amendments will be considered as having been received on time if they are received by the International Bureau after the expiration of the applicable time limit but before the completion of the technical preparations for international publication (Rule 46.1).

#### Where not to file the amendments?

The amendments may only be filed with the International Bureau and not with the receiving Office or the International Searching Authority (Rule 46.2).

Where a demand for international preliminary examination has been its filed, see below.

#### How?

Either by cancelling one or more entire claims, by adding one or more new claims or by amending the text of one or more of the claims as filed.

A replacement sheet must be submitted for each sheet of the claims which, on account of an amendment or amendments, differs from the sheet originally filed.

All the claims appearing on a replacement sheet must be numbered in Arabic numerals. Where a claim is cancelled, no renumbering of the other claims is required. In all cases where claims are renumbered, they must be renumbered consecutively (Administrative Instructions, Section 205(b)).

The amendments must be made in the language in which the international application is to be published.

#### What documents must/may accompany the amendments?

### Letter (Section 205(b)):

The amendments must be submitted with a letter.

The letter will not be published with the international application and the amended claims. It should not be confused with the "Statement under Article 19(1)" (see below, under "Statement under Article 19(1)").

The letter must be in English or French, at the choice of the applicant. However, if the language of the international application is English, the letter must be in English; if the language of the international application is French, the letter must be in French.

The letter must indicate the differences between the claims as filed and the claims as amended. It must, in particular, indicate, in connection with each claim appearing in the international application (it being understood that identical indications concerning several claims may be grouped), whether

- (i) the claim is unchanged;
- (ii) the claim is cancelled;
- (iii) the claim is new;
- (iv) the claim replaces one or more claims as filed;
- (v) the claim is the result of the division of a claim as filed.

## The following examples illustrate the manner in which amendments must be explained in the accompanying letter:

- [Where originally there were 48 claims and after amendment of some claims there are 51]:
   "Claims 1 to 29, 31, 32, 34, 35, 37 to 48 replaced by amended claims bearing the same numbers; claims 30, 33 and 36 unchanged; new claims 49 to 51 added."
- [Where originally there were 15 claims and after amendment of all claims there are 11]: "Claims 1 to 15 replaced by amended claims 1 to 11."
- [Where originally there were 14 claims and the amendments consist in cancelling some claims and in adding new claims]:
   "Claims 1 to 6 and 14 unchanged; claims 7 to 13 cancelled; new claims 15, 16 and 17 added." or
   "Claims 7 to 13 cancelled; new claims 15, 16 and 17 added; all other claims unchanged."
- 4. [Where various kinds of amendments are made]: "Claims 1-10 unchanged; claims 11 to 13, 18 and 19 cancelled; claims 14, 15 and 16 replaced by amended claim 14; claim 17 subdivided into amended claims 15, 16 and 17; new claims 20 and 21 added."

#### "Statement under article 19(1)" (Rule 46.4)

The amendments may be accompanied by a statement explaining the amendments and indicating any impact that such amendments might have on the description and the drawings (which cannot be amended under Article 19(1)).

The statement will be published with the international application and the amended claims.

#### It must be in the language in which the international appplication is to be published.

It must be brief, not exceeding 500 words if in English or if translated into English.

It should not be confused with and does not replace the letter indicating the differences between the claims as filed and as amended. It must be filed on a separate sheet and must be identified as such by a heading, preferably by using the words "Statement under Article 19(1)."

It may not contain any disparaging comments on the international search report or the relevance of citations contained in that report. Reference to citations, relevant to a given claim, contained in the international search report may be made only in connection with an amendment of that claim.

#### Consequence if a demand for international preliminary examination has already been filed

If, at the time of filing any amendments under Article 19, a demand for international preliminary examination has already been submitted, the applicant must preferably, at the same time of filing the amendments with the International Bureau, also file a copy of such amendments with the International Preliminary Examining Authority (see Rule 62.2(a), first sentence).

#### Consequence with regard to translation of the international application for entry into the national phase

The applicant's attention is drawn to the fact that, where upon entry into the national phase, a translation of the claims as amended under Article 19 may have to be furnished to the designated/elected Offices, instead of, or in addition to, the translation of the claims as filed.

For further details on the requirements of each designated/elected Office, see Volume II of the PCT Applicant's



(PCT Article 18 and Rules 43 and 44)

Applicant's or agent's file reference	Applicant's or agent's file reference FOR FURTHER see Notification of Transmittal of International Search Report (Form PCT/ISA/220) as well as, where applicable, item 5 below.					
International application No.	International filing date (day/month/year)	(Earliest) Priority Date (day/month/year)				
PCT/GB 00/02536	30/06/2000	02/07/1999				
Applicant						
NATIONAL POWER PLC et al.						
This International Search Report has bee according to Article 18. A copy is being tr	This International Search Report has been prepared by this International Searching Authority and is transmitted to the applicant according to Article 18. A copy is being transmitted to the International Bureau.					
This International Search Report consists  It is also accompanied by	of a total of Sheets.  a copy of each prior art document cited in this	s report.				
Basis of the report      With regard to the lenguage, the	international search was carried out on the ba	isis of the international application in the				
language in which it was filed, un	less otherwise indicated under this item.	,				
the international search w Authority (Rule 23.1(b)).	vas carried out on the basis of a translation of	the international application furnished to this				
was carried out on the basis of th  contained in the internation		nternational application, the international search				
furnished subsequently to	this Authority in written form.					
	furnished subsequently to this Authority in computer readble form.					
the statement that the sui international application a	bsequently furnished written sequence listing of the s	does not go beyond the disclosure in the				
the statement that the inf furnished.	ormation recorded in computer readable form	is identical to the written sequence listing has been				
2. Certain claims were fou	ind unsearchable (See Box I).					
3. Unity of invention is lac	king (see Box II).					
4. With regard to the <b>title</b> ,						
the text is approved as su	ubmitted by the applicant.					
the text has been established by this Authority to read as follows:						
5. With regard to the abstract,	5. With regard to the abstract.					
the text is approved as submitted by the applicant.						
the text has been established, according to Rule 38.2(b), by this Authority as it appears in Box III. The applicant may, within one month from the date of mailing of this international search report, submit comments to this Authority.						
as suggested by the app	licant.	None of the figures.				
X because the applicant fair	because the applicant failed to suggest a figure.					
because this figure better characterizes the invention.						



International	Application No
T/GB	00/02536

A. CL	ASSIFICATION	OF SUBJECT	MATTER	
IPC	7 HO1M	8/04	H01M8	/18

According to International Patent Classification (IPC) or to both national classification and IPC

#### B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols) IPC 7 H01M

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

PAJ, EPO-Internal, CHEM ABS Data, INSPEC, COMPENDEX

Category °	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	WO 94 09522 A (NAT POWER PLC ;ZITO RALPH (US)) 28 April 1994 (1994-04-28) claims 1-3,6,7,9,10 page 15, line 15 -page 16, line 31; figure 3A page 18, line 16 - line 35 page 25, line 19 -page 26, line 14	1-19
A	US 5 612 148 A (ZITO RALPH) 18 March 1997 (1997-03-18) column 3, line 66 -column 4, line 27 column 7, line 21 - line 45; claims 1,2,8,10; figure 2	1-19
A	WO 94 09526 A (NAT POWER PLC ;ZITO RALPH (US)) 28 April 1994 (1994-04-28) claims 1,5,6,8,10 page 11, line 1 - line 29; figure 1E	1-19
X Furth	ner documents are listed in the continuation of box C.  X Patent family member	s are listed in annex.

· ·	-/
X Further documents are listed in the continuation of box C.	Patent family members are listed in annex.
Special categories of cited documents:      "A" document defining the general state of the art which is not considered to be of particular relevance      "E" earlier document but published on or after the international filling date      "L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)      "O" document referring to an oral disclosure, use, exhibition or other means      "P" document published prior to the international filling date but later than the priority date claimed	<ul> <li>"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention</li> <li>"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone</li> <li>"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.</li> <li>"&amp;" document member of the same patent family</li> </ul>
Date of the actual completion of the international search	Date of mailing of the international search report
17 October 2000	24/10/2000
Name and mailing address of the ISA	Authorized officer
European Patent Office, P.B. 5818 Patentlaan 2 NL – 2280 HV Rijswijk Tel. (+31-70) 340-2040, Tx. 31 651 epo nl, Fax: (+31-70) 340-3016	Battistig, M



Linternational Application No CT/GB 00/02536

(Continui	Ition) DOCUMENTS CONSIDERED TO BE RELEVANT	
Category °	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
1	WO 94 09525 A (NAT POWER PLC ;ZITO RALPH (US)) 28 April 1994 (1994-04-28) claims 1-9	1-19
<b>\</b>	DE 35 22 714 A (FRAUNHOFER GES FORSCHUNG) 8 January 1987 (1987-01-08) claims 1,3,7	1-19
	<b></b>	
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mation on patent family members

International Application No CT/GB 00/02536

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
WO 9409522 A	28-04-1994	AT 139372 T AU 671863 B AU 5153593 A BG 61627 B BG 99562 A BR 9307234 A CA 2145883 A CN 1087753 A,B CZ 9500954 A DE 69303179 D DE 69303179 T DK 664929 T EG 20194 A EP 0664929 A ES 2089853 T FI 951815 A GR 3020714 T HK 1001071 A HU 72141 A,B IL 107236 A JP 8502385 T NO 951377 A NZ 256709 A PL 308266 A RU 2110118 C SG 52426 A SK 48195 A US 5439757 A ZA 9307283 A	15-06-1996 12-09-1996 09-05-1994 30-01-1998 30-11-1995 13-10-1999 28-04-1994 08-06-1994 16-08-1995 18-07-1996 10-10-1996 28-10-1997 02-08-1995 01-10-1996 13-06-1995 30-11-1996 22-05-1998 28-03-1996 31-12-1995 12-03-1996 07-04-1995 27-11-1995 24-07-1995 27-04-1998 28-09-1998 13-09-1995 08-08-1995 30-03-1995
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WO 9409526 A	28-04-1994	AT 152860 T AT 162338 T AU 672049 B AU 5153793 A AU 676691 B AU 5153893 A CN 1087752 A,B DE 69310529 D DE 69310529 T DE 69316387 D DE 69316387 T DK 664931 T DK 664931 T DK 664932 T EG 20201 A EP 0664931 A EP 0664931 A EP 0664932 A ES 2104179 T ES 2111774 T W0 9409525 A GR 3024385 T GR 3026051 T HK 1007461 A IL 107237 A JP 8502388 T JP 8502388 T US 5496659 A	15-05-1997 15-01-1998 19-09-1996 09-05-1994 20-03-1997 09-05-1994 08-06-1994 12-06-1997 06-11-1997 19-02-1998 28-05-1998 08-12-1997 14-09-1998 30-10-1997 02-08-1995 02-08-1995 01-10-1997 16-03-1998 28-04-1994 28-11-1997 29-05-1998 09-04-1999 14-05-1996 12-03-1996 05-03-1996

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CT/GB 00/02536 Patent family **Publication** Patent document Publication date cited in search report member(s) 30-03-1995 WO 9409526 ZA 9307284 A Α n 15-05-1997 28-04-1994 AT WO 9409525 Α 152860 15-01-1998 162338 AT T 19-09-1996 ΑU 672049 B 09-05-1994 ΑU 5153793 A 20-03-1997 ΑU 676691 B ΑU 5153893 A 09-05-1994 08-06-1994 CN 1087752 A,B 12-06-1997 DE 69310529 D DE 69310529 06-11-1997 DE 69316387 D 19-02-1998 DE 69316387 T 28-05-1998 08-12-1997 DK 664931 T DK 664932 T 14-09-1998 EG 20201 A 30-10-1997 EP 0664931 A 02-08-1995 EP 0664932 02-08-1995 ES 2104179 T 01-10-1997 ES 2111774 T 16-03-1998 WO 9409526 A 28-04-1994 GR 3024385 T 28-11-1997 GR 3026051 T 29-05-1998 HK 1007461 A 09-04-1999 IL 107237 A 14-05-1996 JP 8502387 12-03-1996 JP 8502388 T 12-03-1996 US 5496659 A 05-03-1996

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30-03-1995

06-06-1995

Form PCT/ISA/210 (patent family annex) (July 1992)

DE 3522714

Α

08-01-1987

International Application No

PCT

## INTERNATIONAL PRELIMINARY EXAMINATION REPORT

(PCT Article 36 and Rule 70)

Applicant's	s or agent's file reference		
SJA/532	•	FOR FURTHER ACTION	See Notification of Transmittal of International Preliminary Examination Report (Form PCT/IPEA/416)
Internation	al application No.	International filing date (day/month	//year) Priority date (day/month/year)
PCT/GB	00/02536	30/06/2000	02/07/1999
Internation H01M8/0	al Patent Classification (IPC) or no 04	ational classification and IPC	•
NATION	AL POWER PLC et al.		
	international preliminary exam s transmitted to the applicant		by this International Preliminary Examining Authority
2. This	REPORT consists of a total of	f 4 sheets, including this cover s	neet.
t	een amended and are the ba	ed by ANNEXES, i.e. sheets of th sis for this report and/or sheets o 07 of the Administrative Instructi	e description, claims and/or drawings which have ontaining rectifications made before this Authority ons under the PCT).
	e annexes consist of a total of		
3. This	report contains indications rela	ating to the following items:	Č
1	☑ Basis of the report		
II	☐ Priority		
111	☐ Non-establishment of o	opinion with regard to novelty, inv	entive step and industrial applicability
IV	Lack of unity of inventi		
V	Reasoned statement u citations and explanation	nder Article 35(2) with regard to one suporting such statement	novelty, inventive step or industrial applicability;
VI	☐ Certain documents cit		
VII	☐ Certain defects in the in	nternational application	
VIII	☐ Certain observations o	n the international application	
Date of sub	Date of submission of the demand  Date of completion of this report		completion of this report
29/01/20	01	04.05.20	01
	mailing address of the internationa	al Authoriz	ed officer
preliminary	examining authority: European Patent Office		Establish The State of the Stat
0)))	D-80298 Munich	Fitzpat	rick, J
Tel. +49 89 2399 - 0 Tx: 523656 epmu d Fax: +49 89 2399 - 4465			19 No. +49 89 2399 8570



# INTERNATIONAL PRELIMINARY EXAMINATION REPORT

International application No. PCT/GB00/02536

i. Basi	is of	the	rep	oort
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1.	the and	receiving Office in	ments of the international application (Replacement sheets which have been furnished to response to an invitation under Article 14 are referred to in this report as "originally filed" to this report since they do not contain amendments (Rules 70.16 and 70.17)):				
	1-2	21	as originally filed				
	Cla	nims, No.:					
	1-1	9	as originally filed				
	Dra	awings, sheets:					
	1/8	-8/8	as originally filed				
2.	Wit lang	h regard to the <b>lang</b> guage in which the i	nuage, all the elements marked above were available or furnished to this Authority in the nternational application was filed, unless otherwise indicated under this item.				
	The	ese elements were a	available or furnished to this Authority in the following language: , which is:				
		the language of a	translation furnished for the purposes of the international search (under Rule 23.1(b)).				
		the language of pu	blication of the international application (under Rule 48.3(b)).				
		the language of a t 55.2 and/or 55.3).	translation furnished for the purposes of international preliminary examination (under Rule				
3.	Witi inte	n regard to any <b>nuc</b> rnational preliminan	leotide and/or amino acid sequence disclosed in the international application, the yexamination was carried out on the basis of the sequence listing:				
		contained in the in	ternational application in written form.				
		filed together with	the international application in computer readable form.				
		furnished subsequ	ently to this Authority in written form.				
		l furnished subsequently to this Authority in computer readable form.					
			the subsequently furnished written sequence listing does not go beyond the disclosure in oplication as filed has been furnished.				
		The statement that listing has been fur	the information recorded in computer readable form is identical to the written sequence nished.				
4.	The	amendments have	resulted in the cancellation of:				
		the description,	pages:				
		the claims,	Nos.:				

# INTERNATIONAL PRELIMINARY EXAMINATION REPORT

International application No. PCT/GB00/02536

		the drawings,	sheets:						
5.	☐ This report has been established as if (some of) the amendments had not been made, since they have bee considered to go beyond the disclosure as filed (Rule 70.2(c)):								
		(Any replacement she report.)	eet contai	ning such	amendments	s must be re	eferred to unde	r item 1 an	d annexed to this
6.	Add	litional observations, if	necessar	y:					
V.		Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement							
1.	Stat	ement							
	Nov	relty (N)	Yes: No:	Claims Claims	1-19				
	Inve	entive step (IS)	Yes: No:	Claims Claims	1-19				
	Indu	strial applicability (IA)	Yes: No:	Claims Claims	1-19				
_									

2. Citations and explanations see separate sheet

# **Section V.2: Citations and Explanations**

D1 = WO 94 09522

D2 = US 5 612 148

With particular respect to the specific passages cited in the International Search Report (ISR), although documents D1 and D2 respectively use external electrochemical cells for the oxidation of halide and sulfide or bisulfide, neither these nor the remaining documents of the ISR anticipate or fairly suggest the method of rebalancing the electrolytes of part (iii) of current claim 1. Moreover, none of the cell systems of the prior art would be suitable for carrying out said method. In particular, taking document D1 as closest state of the art, on the basis of it's disclosure, the genuine apparatus features of current main claim 11 are distinguished over that of Fig.3A in combination with Fig. 3B or 3C of D1 via:

- the cation exchange membrane of the auxiliary cell.
- the means for circulating an electrolyte through the -ve chamber of the auxiliary cell
- the provision of means for alternatively circulating electrolyte 1 or 2 through the +ve chamber of the auxiliary cell.

These all contribute to ensuring added versatility as well as improved electrolyte rebalancing over the prior art whilst minimising the undesirable reduction of polysulfide to sulfide and oxidation of polysulphide/sulfide to "lost" sulphate. This ensures that the requirements of Art.33(3) PCT are also fulfilled.



(PCT Article 18 and Rules 43 and 44)

Applicant's or agent's file reference	(Form PCT/ISA/220) as well as, where applicable, item 5 below.							
SJA/53278/006 International application No.	International filing date (day/month/year)	(Earliest) Priority Date (day/month/year)						
PCT/GB 00/02536	30/06/2000	02/07/1999						
Applicant								
NATIONAL POWER PLC et al.								
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This International Search Report has bee according to Article 18. A copy is being tr	n prepared by this International Searching Auth ansmitted to the International Bureau.	nority and is transmitted to the applicant						
This International Search Report consists	of a total of sheets.							
	a copy of each prior art document cited in this	report.						
Basis of the report	· · · · · · · · · · · · · · · · · · ·							
	international search was carried out on the bas less otherwise indicated under this item.	sis of the international application in the						
the international search w Authority (Rule 23.1(b)).	vas carried out on the basis of a translation of th	ne international application furnished to this						
b. With regard to any nucleotide ar was carried out on the basis of th	nd/or amino acid sequence disclosed in the interest sequence listing.	ternational application, the international search						
l <del></del>	onal application in written form.							
filed together with the inte	ernational application in computer readable form	n.						
furnished subsequently to	furnished subsequently to this Authority in written form.							
furnished subsequently to	furnished subsequently to this Authority in computer readble form.							
	osequently furnished written sequence listing do is filed has been furnished.	oes not go beyond the disclosure in the						
the statement that the infe furnished	ormation recorded in computer readable form is	sidentical to the written sequence listing has been						
Certain claims were four	nd unsearchable (See Box I).							
3. Unity of Invention is lac	king (see Box II).							
4. With regard to the <b>title</b> ,								
nn .	ibmitted by the applicant.							
	shed by this Authority to read as follows:							
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5. With regard to the abstract,								
	ibmitted by the applicant.							
the text has been established, according to Rule 38.2(b), by this Authority as it appears in Box III. The applicant may, within one month from the date of mailing of this international search report, submit comments to this Authority.								
6. The figure of the <b>drawings</b> to be pub	ished with the abstract is Figure No.	3						
as suggested by the appli	cant.	None of the figures.						
X because the applicant fail	ed to suggest a figure.							
because this figure better characterizes the invention.								

international Application No

A. CLASSIFICATION OF SUBJECT MANUER IPC 7 H01M8/04 H01M8/18

According to International Patent Classification (IPC) or to both national classification and IPC

#### B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols) IPC 7 - H01M

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

PAJ, EPO-Internal, CHEM ABS Data, INSPEC, COMPENDEX

Category °	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	WO 94 09522 A (NAT POWER PLC ;ZITO RALPH (US)) 28 April 1994 (1994-04-28) claims 1-3,6,7,9,10 page 15, line 15 -page 16, line 31; figure 3A	1–19
	page 18, line 16 - line 35 page 25, line 19 -page 26, line 14	
A	US 5 612 148 A (ZITO RALPH) 18 March 1997 (1997-03-18) column 3, line 66 -column 4, line 27 column 7, line 21 - line 45; claims 1,2,8,10; figure 2	1-19
A	WO 94 09526 A (NAT POWER PLC ;ZITO RALPH (US)) 28 April 1994 (1994-04-28) claims 1,5,6,8,10 page 11, line 1 - line 29; figure 1E	1-19

Y Further documents are listed in the continuation of box C.	Patent family members are listed in annex.
<ul> <li>Special categories of cited documents:</li> <li>"A" document defining the general state of the art which is not considered to be of particular relevance</li> <li>"E" earlier document but published on or after the international filing date</li> <li>"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)</li> <li>"O" document referring to an oral disclosure, use, exhibition or other means</li> <li>"P" document published prior to the international filing date but later than the priority date claimed</li> </ul>	"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention  "X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone  "Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.  "&" document member of the same patent family
Date of the actual completion of the international search	Date of mailing of the international search report
17 October 2000	24/10/2000
Name and mailing address of the ISA	Authorized officer
European Patent Office, P.B. 5818 Patentlaan 2 NL – 2280 HV Rijswijk Tel. (+31-70) 340-2040, Tx. 31 651 epo nl, Fax: (+31-70) 340-3016	Battistig, M

International Application No
T/GB 00/02536

Category °	ation) DOCUMENTS CONSIDERED TO BE RELEVANT  Citation of document, with indication, where appropriate, of the relevant passages	Delevent to dain No.
Category	Grador or document, with indication, where appropriate, or the relevant passages	Relevant to claim No.
A	WO 94 09525 A (NAT POWER PLC ;ZITO RALPH (US)) 28 April 1994 (1994-04-28) claims 1-9	1-19
A	DE 35 22 714 A (FRAUNHOFER GES FORSCHUNG) 8 January 1987 (1987-01-08) claims 1,3,7	1-19
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AT

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Information on patent family members

**Publication** 

date

28-04-1994

Patent document

cited in search report

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WO 9409522

US

WO

International Application No T/GB 00/02536 Publication Patent family member(s) date 139372 T 15-06-1996 671863 B 12-09-1996 5153593 A 09-05-1994 61627 B 30-01-1998 99562 A 30-11-1995 9307234 A 13-10-1999 2145883 A 28-04-1994 1087753 A,B 08-06-1994 9500954 A 16-08-1995 69303179 D 18-07-1996 69303179 T 10-10-1996 664929 T 28-10-1996 20194 A 30-10-1997 0664929 A 02-08-1995 2089853 T 01-10-1996 951815 A 13-06-1995 3020714 T 30-11-1996 1001071 A 22-05-1998 72141 A,B 28-03-1996 31-12-1995 107236 A 12-03-1996 8502385 Τ 951377 A 07-04-1995 256709 A 27-11-1995

				PL RU SG SK US ZA	308266 2110118 52426 48195 5439757 9307283	C A A	24-07-1995 27-04-1998 28-09-1998 13-09-1995 08-08-1995 30-03-1995
}	5612148	Α	18-03-1997	NONE			
	9409526	<b>A</b>	28-04-1994	AT AU AU AU AU AU DE DE DE DE DE DE ES WO GR HLL JP US	672049 5153793 676691 5153893 1087752 69310529 69310529 69316387 69316387 664931 664932 20201 0664931 0664932 2104179 2111774 9409525 3024385 3026051 1007461 107237 8502387	T B A B A A B D T D T T T A A A T T A A T T T A A T T T A A T T T A A T T T A A T T T T T T T T T T T T T T T T T T T T	15-05-1997 15-01-1998 19-09-1996 09-05-1994 20-03-1997 09-05-1994 08-06-1997 06-11-1997 19-02-1998 28-05-1998 08-12-1997 14-09-1998 30-10-1997 02-08-1995 01-10-1997 16-03-1998 28-04-1994 28-11-1997 29-05-1998 09-04-1999 14-05-1996 12-03-1996 05-03-1996

International Application No mation on patent family members /GB 00/02536 **Publication** Patent document Publication Patent family cited in search report date member(s) date WO 9409526 Α ZA 9307284 A 30-03-1995 Α 28-04-1994 WO 9409525 AT 15-05-1997 152860 T AT 162338 T 15-01-1998 672049 B ΑU 19-09-1996 ΑU 5153793 A 09-05-1994 AU 676691 20-03-1997 ΑU 5153893 A 09-05-1994 CN 1087752 A,B 08-06-1994 DE 69310529 D 12-06-1997 DE 69310529 T 06-11-1997 DE 69316387 D 19-02-1998 DE 69316387 T 28-05-1998 DK 664931 T 08-12-1997 DK 664932 T 14-09-1998 EG 20201 A 30-10-1997 EP 0664931 A 02-08-1995 EP 0664932 A 02-08-1995 2104179 T ES 01-10-1997 ES 2111774 T 16-03-1998 WO 9409526 A 28-04-1994 GR 3024385 T 28-11-1997 GR 3026051 T 29-05-1998 HK 09-04-1999 1007461 A IL 107237 A 14-05-1996 JP 8502387 T 12-03-1996

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